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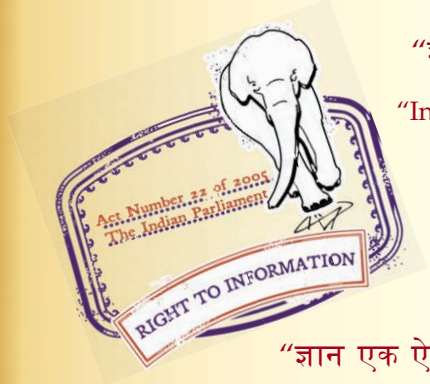
IS 4027-11 (2000): Chemical Analysis of Bronzes - Methods, Part 11: Determination of Lead - Ethylenediamine Tetra-acetic Acid (EDTA) - Titrimetric Method [MTD 8: Copper and Copper Alloys]



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"Knowledge is such a treasure which cannot be stolen"

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IS 4027 (Part 11) : 2000
(Reaffirmed 2006)

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कांसे के रासायनिक विश्लेषण — पद्धतियाँ

भाग 11 सीसा ज्ञात करना — इथायलीनडाईअमाईन टेट्राएसिटिक ऐसिड (इ डी टी ए) — टाईट्रीमिट्रिक पद्धति

(पहला पुनरीक्षण)

Indian Standard

CHEMICAL ANALYSIS OF BRONZES — METHODS

PART 11 DETERMINATION OF LEAD — ETHYLENEDIAMINE TETRAACETIC ACID (EDTA) — TITRIMETRIC METHOD

(First Revision)

ICS 77.120.30;77.120.60

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

February 2000

Price Group 1

FOREWORD

This Indian Standard (Part 11) (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical/Instrumental Analysis of Non-ferrous Methods Sectional Committee had been approved by the Metallurgical Engineering Division Council.

IS 4027 was first published in 1967, and covered determination of Cu, Pb, Sn, Mn, P, Ni, Fe, Si, AL, Zn and Sb in bronzes. While reviewing this standard the committee decided that it was convenient to revise it in series of parts which on publication will supersede the relevant method for determination given in IS 4027 : 1967. The other parts in this series are as follows:

Part 1	Determination of copper and lead by electrolytic method
Part 2	Determination of manganese by photometric method
Part 3	Determination of phosphorus by volumetric method
Part 4	Determination of nickel by photometric method
Part 5	Determination of tin by iodimetric method
Part 6	Determination of zinc by complexometric method
Part 7	Determination of antimony by rhodamine B spectrophotometric method
Part 8	Determination of iron
Part 9	Determination of aluminium by atomic absorption spectrophotometric method
Part 10	Determination of silicon

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it should be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

CHEMICAL ANALYSIS OF BRONZES — METHODS

PART 11 DETERMINATION OF LEAD — ETHYLENEDIAMINE TETRAACETIC ACID (EDTA) — TITRIMETRIC METHOD

(First Revision)

1 SCOPE

This standard (Part 11) covers the determination of lead in concentration range from 2.0 to 30.0 percent in copper alloys by EDTA titrimetric method. Determination of lead by electrolytic method is covered in Part 1 of this standard.

2 REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
1817 : 1961	Methods of sampling of non-ferrous metals for chemical analysis

3 SAMPLING

Samples shall be drawn and prepared in accordance with IS 1817.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and water, shall be employed for the test.

5 DETERMINATION OF LEAD BY THE ETHYLENEDIAMINE TETRAACETIC ACID (EDTA) — TITRIMETRIC METHOD

5.1 Outline of the Method

Lead diethyldithiocarbamate is extracted in chloroform from alkaline tartarate cyanide solution. After the removal of organic material, lead is titrated with disodium ethylene-diamine tetraacetic acid (EDTA) solution.

5.2 Reagents

5.2.1 *Fluoroboric Acid* (37 to 40 percent)

5.2.2 *Dilute Nitric Acid* (50 percent) (*v/v*)

5.2.3 *Sodium Tartarate Solution* (250 g/l)

Dissolve 250 g of sodium tartarate dihydrate in water and dilute to 1 litre.

5.2.4 *Sodium Hydroxide Solution* (250 g/l)

Dissolve 250 g of sodium hydroxide (NaOH) in water and dilute to 1 litre. Store in a plastic bottle.

5.2.5 *Sodium Cyanide Solution* (200 g/l)

Dissolve 200 g of sodium cyanide (NaCN) in water and dilute to 1 litre.

WARNING — Sodium cyanide is extremely poisonous and extreme care must be exercised in handling and disposal. Aqueous solutions containing cyanide must be added to a reservoir containing $FeSO_4$ to convert CN^- to $Fe(CN)_6^{4-}$. No acid must ever be added to avoid formation of poisonous HCN.

5.2.6 *Sodium Diethyl dithiocarbam ate Solution* (100 g/l)

Dissolve 10 g of sodium diethyldithiocarbamate in water and dilute to 100 ml. Freshly prepared solution should be used.

5.2.7 *Chloroform*

5.2.8 *Ascorbic Acid*

5.2.9 *Sodium Hydroxide Solution* (250 g/l)

Dissolve 250 g of sodium hydroxide (NaOH) in water and dilute to 1 litre. Store in a plastic bottle.

5.2.10 *Hexamethylenetetramine*

5.2.10.1 *Disodium ethylene-diamine tetraacetic acid (EDTA) standard solution (0.025 M)*

Dissolve 9.3 g of disodium ethylene-diamine tetraacetate dihydrate in water, transfer to a 1-litre volumetric flask, dilute to volume and mix.

Standardization — Transfer 25 ml of lead solution (1 ml= 6.0 mg Pb) to a 25-ml beaker and dilute

to 100 ml. Proceed as directed in **5.3.7**. Calculate the lead equivalent of the solution as follows:

$$\text{Lead equivalent, g/ml} = A/B$$

where

A = weight, in g, of lead; and

B = volume, in ml, of EDTA solution required for titration of lead solution.

5.2.11 *Xylenol Orange*

5.2.12 *Lead, Standard Solution* (1 ml = 6.0 mg Pb)

Transfer 1.500 g of lead (purity 99.9 percent minimum) to a 150 ml beaker. Add 10 ml of dilute nitric acid (50 percent) and heat until dissolution is complete. Boil to remove oxides of nitrogen, cool, transfer to a 250 ml volumetric flask, dilute to volume, and mix.

5.2.13 *Disodium (Ethylene-diamine Tetraacetic Acid) (EDTA) Standard Solution* (0.025 M)

Dissolve 9.3 g of disodium ethylene-diamine tetracetate dihydrate in water, transfer to a 1-litre volumetric flask, dilute to volume, and mix.

Standardization — Transfer 25 ml of lead solution (1 ml = 6.0 mg Pb) in a 250-ml beaker and dilute to 100 ml. Proceed as directed in **5.3.7**. Calculate the lead equivalent of the solution as follows:

$$\text{Lead equivalent, g/ml} = A/B$$

where

A = weight, in g, of lead; and

B = volume, in ml, of EDTA solution required for titration of the lead solution.

5.3 Procedure

5.3.1 Weigh 1.00 g of sample containing lead content between 2.0 to 20.0 percent and 0.60 g of sample containing lead content between 20.0 to 30.0 percent and transfer to a 250-ml beaker.

5.3.2 Add 5 ml of fluoroboric acid (37 to 40 percent) and then 10 ml of dilute nitric acid (50 percent). Cover the beaker and heat until dissolution is complete. Boil until oxides of nitrogen have been expelled and then cool.

5.3.3 Wash the cover and walls of the beaker. Add 25 ml of sodium tartarate solution, 25 ml of sodium *hydroxide solution* and 25 ml of sodium cyanide solution, mixing after each addition. Cool to room temperature.

NOTE — Sodium cyanide is extremely poisonous. Use measuring cylinder for its addition.

5.3.4 Transfer to a 250-ml separatory funnel. Add 15 ml of sodium diethyldithiocarbamate solution and 15 ml of chloroform, and shake for 30 s. Allow the layers to separate, draw off the lower organic layer into a 250-ml beaker, retaining the aqueous layer. Add 5 ml more of diethyldithiocarbamate solution to the separatory funnel and mix. If no precipitate forms proceed as directed in **5.3.5**. If a precipitate does form, add 5 ml of diethyldithiocarbamate solution and 10 ml of chloroform, shake for 30 s and draw off the organic layer into the 250-ml beaker containing the extract.

5.3.5 Extract twice with additional 10 ml portions of chloroform, adding the extracts to the extracts reserved in **5.3.4**.

5.3.6 Add 10 ml of dilute hydrochloric acid (50 percent) to the combined extracts and place on a hot plate. Cover the beaker with a raised cover glass, and evaporate the solution to a volume of 2 to 4 ml. Wash the cover and walls of the beaker, dilute to 100 ml, and heat to dissolve salts.

5.3.7 Place the beaker on a magnetic stirrer and stir. Add 10 to 20 mg of ascorbic acid and 3 or 4 drops of xylenol orange solution. Add enough hexamethylenetetramine to colour the solution purple. Add 4 or 5 drops of sodium cyanide solution and titrate with the EDTA solution. When a yellow colour begins to appear, stop the titration and add 2 to 3 g of hexamethylenetetramine and a drop of xylenol orange solution. Titrate dropwise until the colour changes from purplish-red to yellow.

5.4 Calculations

5.4.1 Calculate the percentage of lead as follows:

$$\text{Lead, percent by mass} = \frac{C \times D \times 100}{E}$$

where

C = volume in ml, of standard EDTA solution;

D = lead equivalent of EDTA solution, g/ml, and;

E = weight, in g, of sample used.

NOTE — The waste generated in analysis contains sodium cyanide which is extremely poisonous. Sufficient care must be exercised in its use and disposal. Such solution should be made alkaline before disposal. It is considered safe to convert CN^- to $Fe(CN)_{64-}$ by addition of $FeSO_4$ before disposal. Any accidental spillage must be flushed with plenty of water. If facilities for safe handling and disposal of sodium cyanide are not available.

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

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Amendments Issued Since Publication

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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110 002
Telephones : 323 01 31, 323 33 75, 323 94 02

Telegrams : Manaksanstha
(Common to all offices)

Regional Offices :		Telephone
Central	: Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110 002	323 76 17, 323 38 41
Eastern	: 1/14 C. I.T. Scheme VII M, V. I. P. Road, Kankurgachi, CALCUTTA 700 054	337 84 99, 337 85 61, 337 86 26, 337 91 20
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